

## Thermodynamic, Kinetic and Adsorptive Parameters of Corrosion Inhibition of Aluminium Using Sorghum bicolor Leaf Extract in H<sub>2</sub>SO<sub>4</sub>

Stephen G. Yiase, Sylvester O. Adejo, Tersoo G. Tyohemba, Ungwanen J. Ahile, and Joseph A. Gbertyo\*

Department of Chemistry, Benue State University,  
Makurdi-Nigeria

\*ja.gbertyo@gmail.com, syiase@bsum.edu.ng

sadejo@bsum.edu.ng, tyogab@gmail.com

ahileuj@gmail.com

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**Abstract:** *The inhibitive action of ethanol leaf extract of Sorghum bicolor has been studied using the weight loss method at the temperature range of 305 K to 315 K. The inhibitive action was observed to increase with increase in concentration of the extract, but decreased with increase in temperature. The decrease in the inhibitive action with rise in temperature is suggestive of physisorption. The evaluated heat of adsorption,  $Q_{ads}$ , ranged from -72.50 kJ/mol to -126.76 kJ/mol and the negative values are indication that the adsorption of inhibitor onto the coupon surface is exothermic. The values of activation energy,  $E_a$ , range from 105.89 to 128.60 kJ/mol in the presence of the extract, clear indication of Chemisorption. Enthalpy of adsorption,  $\Delta H^{\circ}$ , values range from 17.61 kJ/mol to 88.40 kJ/mol with values in the presence of the extract being higher than the free solution. Adsorption of leaf extract of Sorghum bicolor best fitted into the Frumkin, El-Awady isotherms and Adejo-Ekwenchi isotherm models. The decrease in value of parameter  $b$  in Adejo-Ekwenchi isotherm with increase in temperature confirmed that the adsorption mechanism was physisorption.*

**Keywords:** *Corrosion inhibition, aluminium, Sorghum bicolor, adsorption isotherms, physisorption*

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### 1. INTRODUCTION

Aluminium, a light weight metal, is one of the most abundant elements in the Earth's crust. In every million atoms, around 82,000 of them are aluminium, (82,000 ppm), by weight. This is around 8 % or almost one in every 12 atoms. This places aluminium amongst the world's most abundant elements, behind only oxygen and silicon. It finds applications in transport sector where it is used as engine blocks, cylinder heads, and body panels, trucks and buses as sheet and plate for bodies in railway and in aircraft. In construction, aluminium is used in sheet products for roofing and wall cladding, in extrusions for windows and doors, and in castings for builders' hardware. Where as in packaging, aluminium is used in the form of alloy sheet for beverage can bodies and tops, as foil for household and commercial wrap, and in manufactured packaging products such as cartons for fruit juice and packaging for pharmaceuticals. In the electrical sector, aluminium is used in the form of wire, normally reinforced with steel to form cables [1-3].

Corrosion is the deterioration of materials by chemical interaction with their environment. The term corrosion is sometimes applied to the degradation of plastics, concrete and wood, but in most cases refers to metals [3-5]. The use of inhibitors for the control of corrosion of materials, which are in contact with aggressive environment, is an accepted practice. Corrosion inhibitors are substances, which when added in small concentrations to corrosive media decrease or prevent the reaction of the metal with the media. Corrosion protection by inhibitors has been employed in many systems namely; cooling system, refinery units, chemicals, oil and gas production units' boiler, etc. [6-10]. Inhibitors reduce the corrosion rate in a number of ways: by modifying the corrosion potential, retarding the cathodic or anodic corrosion reactions via polarization or passivation of the metal surface, etc. Organic inhibitors have been found to function by adsorption of their ions or molecules onto metal surface. Accurate elucidation of the mechanism of

adsorption of the inhibitor on the metal surface is essential for the design and development of new corrosion inhibitors as well as for a detailed understanding of mechanism of the inhibition processes [11, 12]. Corrosion studies might give insight into its kinetics so as to enable the prediction of the service life of equipment thereby indicating the environmental control for which a particular metal may be satisfactorily used [13, 14]. The present work was designed as a contribution to the growing interest on environmentally benign corrosion inhibitors to study (i) corrosion inhibition of aluminium in 2 M H<sub>2</sub>SO<sub>4</sub> solutions by extracts of *Sorghum bicolor* using weight loss method at a temperature range of 305 K to 315 K, (ii) to evaluate the activation energy, heat of adsorption process as well as determining the best adsorption isotherm for the adsorption of the extracts of *sorghum bicolor* onto the aluminium surface.

## 2. METHODS

### 2.1 Material Preparation

Aluminium wires of 99.8% were obtained from the market and taken to Metallurgical Laboratory of the Department of Mechanical Engineering, University of Agriculture, Makurdi, where the wires were melted in a furnace and casted into sheet. The sheets were mechanically cut into smaller units of 2.1 x 2.1 x 0.35 cm dimension. The coupons were filed using sand paper to ensure smooth surface.

The extract of *Sorghum bicolor* was obtained via cool maceration by soaking the dried leaves in absolute ethanol for 48 hours, with occasional shaking and filtered, thereafter. The ethanol was evaporated over a thermostated water bath at about 351K. The residue, an oily paste was preserved in a desiccator (to avoid contamination) [3, 6, 15].

### 2.2 Weight Loss Measurement

2 M H<sub>2</sub>SO<sub>4</sub> was prepared and used to get a blank for the corrosion inhibition process. Aluminium coupons were suspended into a 50 mL beaker containing 2 M H<sub>2</sub>SO<sub>4</sub> in such a way that the coupons was completely immersed in the solution but held suspended by a thin thread tied to a retort stand. The coupons were removed at intervals of 8 hours and rinsed in ammonium acetate solution to quench the reaction. They were then washed with acetone, dried in a desiccator to cool to ambient temperature, and weighed. Each value used was an average of three measurements [16-20].

*Sorghum bicolor* leaf extract so obtained was used to prepare solutions, first by dissolving in ethanol, containing 5 mL, 10 mL, 15 mL, 20 mL and 25 mL in 1 dm<sup>3</sup> of 2 M H<sub>2</sub>SO<sub>4</sub> respectively. The weight loss, *W<sub>l</sub>*, of aluminium coupons were evaluated in grams as the difference in weight of the coupons before and after immersion in 2 M H<sub>2</sub>SO<sub>4</sub>.

$$Wl = W_i - W_f \quad (1)$$

Where *W<sub>1</sub>* and *W<sub>2</sub>* are initial and final weights of coupon

The inhibitor efficiency, % *IE*, of the extract was calculated using the formula.

$$IE\% = \left(1 - \frac{W_{inh}}{W_{blank}}\right) \times 100 \quad (2)$$

Where *W<sub>inh</sub>* and *W<sub>blank</sub>* are weight losses in the absence and presence of inhibitor, respectively, in the acid solution at 305 K, 307 K, 309 K, 311 K, 313 K and 315 K, respectively.

The degree of surface coverage, *θ*, is given by equation,

$$\theta = 1 - \frac{W_{inh}}{W_{blank}} \quad (3)$$

The corrosion rate, *CR*, of the metal was determined for the immersion period from the results of the weight loss

$$CR \text{ (mgcm}^{-2}\text{h}^{-1}\text{)} = \frac{W_l}{At} \quad (4)$$

Where *W<sub>l</sub>* is the weight loss in milligrams (mg), *A* the coupon surface area in cm<sup>2</sup> and *t* the immersion time in hours.

### 3. RESULTS AND DISCUSSION

#### 3.1 Effect of concentration and temperature on the corrosion of aluminum in 2 M H<sub>2</sub>SO<sub>4</sub>

The effect of temperature on the inhibitive action of extract of leaves of *Sorghum bicolor* on the corrosion of aluminium was determined at various concentrations at the temperature range of 303 K to 315 K. The data (Table 1) reveal that, as the concentration of the inhibitor increased the corrosion rate decreased at all temperatures, showing the propensity of the extract to inhibit the corrosion of the metal [21, 22]. However, the corrosion rate was observed to increase with increase in temperature for a given concentration of the extract.

**Table 1.** Variation of weight loss and corrosion rate with the concentration and temperature for the immersion period

Conc. (mL/L)	Weight loss (g)						CR (mgcm <sup>-2</sup> h <sup>-1</sup> )					
	305K	307K	309K	311K	313K	315K	305K	307K	309K	311K	313K	315K
Blank	0.008	0.013	0.017	0.020	0.023	0.031	0.0850	0.1381	0.1807	0.2126	0.2445	0.3295
5	0.007	0.012	0.016	0.019	0.022	0.030	0.0744	0.1276	0.1701	0.2020	0.2338	0.3189
10	0.006	0.010	0.014	0.018	0.021	0.029	0.0638	0.1063	0.1488	0.1913	0.2232	0.3082
15	0.005	0.009	0.012	0.016	0.019	0.027	0.0532	0.0957	0.1276	0.1701	0.2020	0.2870
20	0.004	0.008	0.011	0.014	0.017	0.025	0.0425	0.0850	0.1169	0.1488	0.1807	0.2657
25	0.004	0.007	0.013	0.013	0.015	0.024	0.0425	0.0744	0.1381	0.1382	0.1894	0.2551

Values of % IE obtained through equation (2) are presented in Table 2. From the Table it is obvious that the inhibition efficiency increased with increase in the extract concentration [2]. However, the decrease in inhibition efficiency is not quite significant. The inhibition efficiency was found to decrease with rise in temperature. The decrease in inhibition efficiency with rise in temperature is actually suggestive of physical adsorption mechanism [23, 24].

In the study of corrosion of aluminium in 2 M H<sub>2</sub>SO<sub>4</sub> solutions, weight at time t designated W<sub>t</sub> when log of W<sub>t</sub> was plotted against time, a linear variation was observed, which confirmed a first order reaction kinetics:

$$\log W = \log W_0 - kt \quad (5)$$

Where W<sub>0</sub> is the weight before immersion, k is the rate constant and t is the time. The values of the rate constant, k, obtained from the slopes of the plots are presented in Table 3.

The results obtained from table 3 from the rate constant values, the half-life values; t<sub>1/2</sub> of the metal in the test solution was calculated using: [25].

$$t_{1/2} = \frac{0.693}{k} \quad (6)$$

Table 3 shows the values of k and t<sub>1/2</sub> for the corrosion reaction in the presence and absence of the inhibitor. It can be seen that the k value increases with increase in temperature at all concentrations, but decreases with increase in the inhibitor concentration. However, t<sub>1/2</sub> values are opposite of k values, that is it decreases with rise in temperature and increases with increase in concentration. The increase in t<sub>1/2</sub> values with increase in concentration shows that the corrosion reaction slowed down when more inhibitor was added [26].

**Table 2.** Percentage inhibition efficiency in 2 M H<sub>2</sub>SO<sub>4</sub> at different temperatures.

Conc. (mL/L)	% IE					
	305K	307K	309K	311K	313K	315K
5	12.50	7.69	5.880	5.00	4.35	3.220
10	25.00	23.08	17.65	10.00	8.70	6.450
15	37.50	30.77	29.41	20.00	17.39	12.90
20	50.00	38.46	35.29	30.00	26.09	19.90
25	50.00	46.15	23.53	35.00	34.78	22.58

**Table 3.** Values of rate constant, k, and half-life (t<sub>1/2</sub>) of the corrosion reaction

Conc. mL/L	k x 10 <sup>-4</sup> (h <sup>-1</sup> )						t <sub>1/2</sub> x10 <sup>3</sup> (h)					
	305K	307K	309K	311K	313K	315K	305K	307K	309K	311K	313K	315
Blank	1.348	2.377	2.509	2.901	3.352	4.380	5.141	2.915	2.762	2.389	2.067	1.582
5	1.229	2.050	2.258	2.781	2.796	4.556	5.638	3.380	3.069	2.492	2.479	1.521
10	1.157	1.647	2.173	2.597	2.665	4.767	5.990	4.208	3.189	2.668	2.600	1.454
15	0.788	1.508	1.912	2.548	2.663	4.563	8.794	4.595	3.624	2.720	2.602	1.519
20	0.808	1.349	1.895	2.519	2.564	4.801	8.577	5.137	3.657	2.751	2.703	1.443
25	0.765	1.188	2.075	1.958	2.433	3.882	12.588	5.833	3.340	3.539	2.848	1.785

**Table 4.** Activation parameters for the dissolution of aluminium coupon in the presence of Sorghum bicolor extract

Conc. mL/L	E <sub>a</sub> kJ/mol	+ΔH <sup>*</sup> kJ/mol	+ΔS <sup>*</sup> kJ/mol	-Q <sub>ads</sub> kJ/mol	ΔG (kJ/mol)						
					305 K	307K	309K	311K	313K	315K	
Blank	98.88	96.29	0.742								
5	105.89	103.33	1.062	97.04	-1.1693	1.9999	0.9406	1.3900	1.7792	2.6092	
10	118.30	115.72	1.628	126.76	-1.5603	-1.3020	-0.4459	1.2502	1.6580	2.5174	
15	125.19	122.62	1.935	103.45	-2.0227	-1.2699	-1.1120	0.2016	0.6504	1.5755	
20	133.37	132.62	2.387	96.13	-2.5886	-1.4056	-1.0646	-0.4483	0.0537	1.0655	
25	128.60	126.02	2.075	72.50	-2.5886	-1.6420	0.9793	-0.4615	-0.4393	1.1386	

Using the linearised Arrhenius equation (equation 7), values of E<sub>a</sub> was obtained [3, 27].

$$\log CR = \frac{E_a}{2.303RT} + \log A \quad (7)$$

Where E<sub>a</sub> is the apparent activation energy, T is absolute temperature, A is the Arrhenius pre-exponential constant and R is the universal gas constant. E<sub>a</sub> was evaluated from the slope of a plot of logarithms of corrosion rate versus 1/T.

The evaluated values of activation energy are listed in table 4. The data shows that the E<sub>a</sub> values in the presence of different concentrations of the extract are higher than that in the free acid. The decrease in apparent activation energy in the presence of the extract denotes chemical adsorption mechanism, while the reverse is usually attributed to physical adsorption. The higher activation energy implies a slow dissolution of the aluminium metal [28-32].

An alternative formula of Arrhenius equation is:

$$\ln\left(\frac{CR}{T}\right) = \ln\left(\frac{R}{Nh}\right) + \frac{\Delta S^*}{R} - \frac{\Delta H^*}{RT} \quad (8)$$

Where h is plank's constant, N the Avogadro's number, ΔH<sup>\*</sup> is the enthalpy of activation and ΔS<sup>\*</sup> the entropy of activation. A plot of ln CR/T versus 1/T gave a straight line with a slope of -ΔH<sup>\*</sup>/R

and an intercept of  $\ln R/Nh + \Delta S^*/R$ , from where the values of  $\Delta H^*$  and  $\Delta S^*$  were calculated and are listed in Table 4. The positive signs of enthalpies ( $\Delta H^*$ ) reflect the endothermic nature of dissolution process. The shift towards positive value of entropies ( $\Delta S^*$ ) implies that the activated complex in the rate-determining step represents dissociation rather than association, meaning that disordering increases on going from reactants to the activated complex [33, 34]. The values of heat of adsorption  $Q_{ads}$  were calculated using:

$$\log\left(\frac{\theta}{1-\theta}\right) = \log A + \log K - \frac{Q_{ads}}{2.303 R} \left(\frac{1}{T}\right) \quad (9)$$

Where  $\theta$  is the degree of surface coverage, R is the gas constant, T is the absolute temperature, and A is a temperature independent factor. Values of heat of adsorption were obtained from the slope  $\left(\frac{-Q_{ads}}{2.303R}\right)$  of a plot of  $\log\left(\frac{\theta}{1-\theta}\right)$  against  $\frac{1}{T}$  as reported in Table 4 above and the values ranged from -72.50 kJ/mol to -126.76 kJ/mol. The  $Q_{ads}$  values decreased with increase in inhibitor concentration. The negative values are an indication that the adsorption of inhibitor onto the coupon surface is exothermic [3, 34].

The equilibrium constant of adsorption process is related to the free energy of adsorption  $\Delta G_{ads}$  as shown in equation (10).

$$\Delta G = -2.303RT \log(55.5K) \quad (10)$$

where R is the gas constant, T is the temperature, K is the equilibrium constant of adsorption. The values of K were obtained through the equation (11).

$$K = \frac{\theta}{[(1-\theta)C]} \quad (11)$$

Where C is the concentration of the extract; the negative value of  $\Delta G_{ads}$  is a suggestion that the adsorption of *Sorghum bicolor* leaves extract onto the metal surface is a spontaneous process and the adsorbed layer is stable. Usually in the adsorption of free energy involved in a physical process,  $\Delta G_{ads} < -40$  kJ/mol [34].

### 3.2 Adsorption Considerations

Adsorption of organic compounds depends upon the charge and the nature of the metal surface, electronic characteristics of the metal surface, ionic species, temperature of the corrosion reaction and the electrochemical potential at the metal-solution interface [7, 22]. The adsorption process of organic substances involves two types of the possible interaction with the metal surface. The first one is weak undirected interaction due to electrostatic attraction between inhibiting organic ions or dipole and the electrically charged surface of the metal. This interaction is termed physical adsorption. The second type of interaction occurs when there is interaction between the adsorbate and adsorbent. This type of interaction involves charge sharing or charge transfer from adsorbate to the atoms of the metal surface in order to form a coordinate type bond and the interaction is termed chemical adsorption [7, 12].

The adsorption isotherms provide important clues regarding the nature of the metal-inhibitor interaction. Inhibitor molecules adsorb on the metal surface if the interaction between molecules and metal surface is higher than that of the water molecule and the surface [3].

In order to obtain the adsorption isotherm, the degrees of surface coverage,  $\theta$ , for various concentrations was calculated. The isotherms are of the general form;

$$f(\theta, x) \exp(-2a\theta) = KC \quad (12)$$

where  $f(\theta, x)$  is the configurational factor which depends upon physical model and the assumption underlying the derivation of the isotherm.  $\theta$  is the surface coverage degree, C is the inhibition concentration in the bulk solution 'a' is the lateral interaction term describing the molecular interaction in the adsorbed layer and the heterogeneity of the surface, x is the size ratio and K is the adsorption-desorption equilibrium constant.

The correlation coefficient, slopes, and adsorption equilibrium constants obtained from the isotherms' plots are shown in table 5. Using the regression coefficient,  $R^2$ , values it can be seen the adsorption of the extract fitted Frumkin, Freundlich, Temkin and El-Awady adsorption isotherms. It has been argued that the transformation of non-linear isotherm equations to linear forms implicitly alters their error structure and may also violate the error variance and normality assumptions of standard least squares, bringing out some limitations in the use of  $R^2$  for such determination [35].

$K_{ads}$  represents the strength of adsorption between adsorbate and adsorbent [2, 7] Larger values of  $K_{ads}$  imply favourable adsorption and hence better inhibitor efficiency [7]. The fact that the values are not so great is an indication that the adsorption process was not too good, which is manifested the inhibition efficiency values.

The values of parameter  $n$  of the Freundlich isotherm are not close to the ideal value of 0.6 [36] so the adsorption process of this extract cannot be modeled by this isotherm. Values of  $y$  parameter of the El-Awady isotherm are greater than unity, which imply the formation of multilayers of inhibitor molecules on the metal surface [36, 37]. The values of the molecular interaction parameters 'a' obtained from Temkin adsorption isotherm are negative indicative of molecular repulsion in the adsorbed layer of the adsorbate [38]. The adsorption has basic features of physical adsorption, so it cannot be modeled by Temkin isotherm, as it is usually a feature of chemical adsorption. Also the values of  $\alpha$  parameters of the Frumkin isotherm are all negative, indicative of repulsion in the adsorbed layer.

**Table 5.** The Adsorption Parameters

Isotherm	Temperature (K)	$R^2$	Slope	$K_{ads}$		$-\Delta G_{ads}$
Freundlich					n	
	305	0.9804	0.9101	0.0301	0.9101	1.30
	307	0.9622	1.1093	0.0151	1.1093	0.45
	309	0.8010	0.9961	0.0148	0.9961	0.51
	311	0.9879	1.2686	0.0062	1.2686	2.76
	313	0.9897	1.3221	0.0048	1.3221	3.44
	315	0.9878	1.2678	0.0040	1.2678	3.94
El-Awady					y	
	305	0.9837	0.9101	0.0180	1.2886	2.54
	307	0.9832	1.1093	0.0095	1.4140	1.63
	309	0.7902	0.9961	0.0119	1.1827	1.07
	311	0.9838	1.2686	0.0042	1.5091	3.77
	313	0.9821	1.3221	0.0033	1.5503	4.42
	315	0.9857	1.2678	0.0032	1.4099	
Frumkin					$-\alpha$	
	305	0.9878	2.1865	0.0065	2.5178	2.59
	307	0.9694	2.6173	0.0047	3.0138	3.43
	309	0.9611	3.1813	0.0037	3.6633	4.07
	311	0.9632	3.2326	0.0035	3.7223	4.24
	313	0.9519	3.3838	0.0033	3.8960	4.42
	315	0.9557	4.6597	0.0023	5.3656	5.39
Temkin					-a	
	305	0.9675	1.6655	0.2938	1.9178	7.08
	307	0.9918	1.8565	0.2678	2.1378	6.89
	309	0.7154	2.0632	0.2220	2.3758	6.45
	311	0.9195	2.0759	0.1997	2.3904	6.22
	313	0.8898	2.0912	0.1850	2.4080	6.06
	315	0.9193	3.2182	0.1997	3.7058	6.30
Adejo - Ekwonchi			b			
	305	0.9440	0.3780	1.675		11.42
	307	0.9710	0.3200	1.581		11.42

## Thermodynamic, Kinetic and Adsorptive Parameters of Corrosion Inhibition of Aluminium Using *Sorghum bicolor* Leaf Extract in H<sub>2</sub>SO<sub>4</sub>

	309	0.6790	0.1860	1.242		10.87
	311	0.9050	0.2390	1.459		11.36
	313	0.8700	0.2280	1.452		11.42
	315	0.9050	0.1430	1.253		11.11

The use of the newly proposed Adejo-Ekwenchi isotherm can resolve ambiguity usually associated with characterisation of adsorption behaviour of extracts [39]. The *b* parameter clearly shows whether the adsorption process is physisorption or chemisorption. The value of *b* for the adsorption of this extract decreases with rise in temperature, signifying a Physisorption process. In conclusion the adsorption of *Sorghum bicolor* can best be model by El-Awady, Frumkin and Adejo-Ekwenchi isotherms.

### 4. CONCLUSION

The ethanol leaf extract of *Sorghum bicolor* in H<sub>2</sub>SO<sub>4</sub> solution showed moderate inhibitive action against the corrosion of aluminium in H<sub>2</sub>SO<sub>4</sub>. The adsorption on aluminium surface obeyed El-Awady, Frumkin and Adejo-Ekwenchi adsorption isotherms and mechanism of adsorption is physisorption.

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